

Examples**Preparation of Examples 1-3:**

5 A 40 l autoclave is flushed with inert gas (nitrogen). The polymer solution and the catalyst are added (Table 1). After the autoclave has been closed it is pressurised several times with protective gas and then with hydrogen. After the pressure has been released the relevant hydrogen pressure is adjusted and the contents heated, with stirring, to the appropriate reaction temperature. The reaction pressure is kept constant after the hydrogen uptake has commenced.

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After the reaction has ended the polymer solution is filtered. The stabiliser is added, solvent is removed from the product at 240°C and the product is processed further as a granular material (Example 2 and 3, Table 2).

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Table 1

Hydrogenation of polystyrene for the preparation of vinylcyclohexane-based polymers

Ex. no.	Polymer mass ²⁾ kg	Solvent l	Catalyst mass ³⁾ g	Reaction temp. °C	Hydrogen pressure bar	Reaction time h	Degree of hydrogenation ¹⁾ %
1	5.0	25 cyclohexane	625 ³⁾	180	100	24.5	100
2	5.7	15 cyclohexane	625	140	100	29.5	100
		10 methyl-t-butyl ether					
3	4.8	15.1 cyclohexane	625	160	100	27	100
		10.1 methyl-t-butyl ether					

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- 1) Determined by $^1\text{H-NMR}$ spectroscopy
- 2) Polystyrene, type 158 k transparent, $\overline{M}_w = 280000$ g/mol, **absolute** \overline{M}_w (weight-average), BASF AG, Ludwigshafen, Germany
- 3) $\text{Ni/SiO}_2/\text{Al}_2\text{O}_3$, Ni 5136 P, Engelhard, De Meern, Netherlands

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The **relative** molecular weights of the hydrogenated polystyrene are, (measured by GPC in tetrahydrofuran, against polystyrene standard)

Example 1 (comparison): 106 000 g/mole

10 Example 2 (comparison): 161 000 g/mole

Example 3 (according to the invention): 167 000 g/mole

The amount of stabiliser and the relative molecular weights of the vinylcyclohexane-based polymers after extrusion are given in Table 2.

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Table 2

Extrusion of vinylcyclohexane-based polymers

Example		Max. processing temp. °C	Molecular weights ¹⁾ \overline{M}_w 10^3 mol^{-1}	Amount of stabiliser ³⁾ %	Sterically hindered phenol ⁴⁾ %	Phosphite phosphonite ⁴⁾ %	Lactone ⁴⁾ %
1 Comparison	Extrusion	240	67		-	-	-
2 Comparison	Extrusion ²⁾	315	142	0.46	25 (Irganox 1010)	75 (Irgafos 168)	-
3 acc. to the invention	Extrusion ²⁾	335	160	0.40	50 (Irganox 1010)	33 (Irgafos P-EPQ)	17 (HP136)

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- 1) determined as relative molecular weights \overline{M}_w against THF-GC polystyrene standard
- 2) CD injection moulding machine, Netstal Diskjet 600
- 3) based on the starting polymer

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4) based on the stabiliser system

Comparison example 1 shows that without the addition of a stabiliser, a drastic decrease in molecular weight (\overline{M}_w) takes place at a processing temperature of only 240°C. The stabiliser system, which is composed only of a sterically hindered phenol and a phosphorus compound, has the disadvantage that a significant decrease in molecular weight takes place at the high temperatures (>300°C) required for the injection moulding of optical high density storage media (comparison example 2). The stabiliser system according to the invention exhibits no significant decrease in the molecular weight of the polymer at a much higher processing temperature (335°C) and is, therefore, particularly suitable as a stabiliser of vinylcyclohexane-based polymers for the manufacture of optical data storage media at high processing temperatures.